"GELCASTING OF POROUS ALUMINA FOR PARTICULATE FILTERING"

A thesis submitted in the partial fulfilment of the requirements for the degree of

Bachelor of Technology

By,

Ansuman Mishra

(108CR036)

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Under the Guidance of:-

Prof. Arun Chowdhury

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

Certificate

This is to inform that the thesis entitled ***"Gel casting of porous alumina for particulate filtering"*** submitted by ***"Ansuman Mishra"* (108CR036)** in partial fulfilments for the requirement of the award of ***Bachelor of Technology in Ceramic Engineering*** at National Institute of Technology, Rourkela is an authentic work carried out by him under my guidance and supervision.

To the best of my knowledge the matter embodied in this thesis has not been submitted to any university/institute for the awarding of any degree/diploma.

Date:

Prof. Arun Choudhury,

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Rourkela- 769008.

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ABBREVIATION GUIDE

SEM	Scanning Electron Microscopy	Darvan-C	Ammonium Polymethacrylate
AP	Apparent Porosity	**MAM**	Methyl Acrylamide
BD	Bulk Density	**MBAM**	Methyl –Bis-Acrylamide
FS	Flexural Strength	**TEMED**	Tetra Methyl Ethylene Diamine
CCS	Cold Crushing Strength	**PEG**	Poly Ethylene Glycol
CTAB	Cetyl Trimethyl AmmoniumBromide		

ACKNOWLEDGEMENT

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Last but not the least I thank almighty, my parents and near and dear ones for being a strong pillar of emotional and financial support during this endeavour of mine.

A HUMBLE SUBMISSION BEFORE YOU ALL

Ansuman Mishra
(108CR036)

ABSTRACT

Gelcasting has been an effective route to manufacturing dense as well as porous ceramics, this project attempts at fabricating porous alumina bodies in the form of layer by layer addition by surfactant action.

Meanwhile in the process of fabricating the body several steps or algorithms have been taken care of, initiating with slurry stabilisation, where we obtain an optimum pH, zeta potential, dispersant concentration viz **(9.2-66.1mV, 0.5%)** combination along with the optimum solid loading for rheology **(8:3, alumina:water),** we proceed on to fabrication of the gelcasted layers accompanied by a study of porosity introduced in them, solid loading of **(7:3, 1:1 alumina:water)** have been tried at different surfactant levels of **2.5, 5, 7.5% w/w.**

Following the above mentioned steps we have a seamless integration of the layers of varying porosity in different combinations and a mechanical property characterisation of the same which includes compressive and flexural strength analysis is studied.

Finally we prepare the desired porous body with a varying porosity concentration gradient and successfully show its filtering action. Microscopy analysis viz SEM has been done and the individual properties studied. This meanwhile marked the successful attainment of 60% porosity at the lab scale as well as we could manufacture a functionally graded seamlessly integrated monolith where the function is porosity necessary for the desired filtration behaviour.

CHAPTER - 1

INSIGHT

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In this project of mine the intricacies of the process of gel casting has been brought out to the forefront. This process has been targeted at devising or fabricating a porous body and the porosity parameters were played with. This was made possible by varying various levels of surfactants in terms of percentage of the total amount of alumina that was taken in the slurry or briefly the total amount of Alumina used as a solid loading[1].

A high temperature form of alumina typically known as α-alumina (Al_2O_3) was used as the primary precursor material along with a set of acrylamide and bis-acrylamide[2] system which act as monomeric and crosslinking units respectively.

The manufactured slurry was incorporated with porosity by using available surfactants which are commercially available in the market[3]. This porous body was fabricated in the form of small discs casted in normal plastic petri dish. Such type of small discs were casted one upon the other and was allowed to set hence leading to a seamless integration and formation of one monolith. Every single disc shaped layer that was casted had a unique variation in a certain function and the function resulted out to be porosity. Thus leading to a multilayer graded material called as a functionally graded material with varying levels of porosity throughout its structure from head to tail. This has been successfully incorporated for filtration action and the filtration of varying materials starting from normal water to corrosive liquids to variety of other materials[1], typically attributed to the inertness, excellent thermo mechanical and corrosion resistant property of Alumina.

Characterisations primarily are porosity focused; some landmark characterisations for porosity performed being, the conventional apparent porosity& bulk density to get an estimate of the open pores, mercury porosimetry to obtain the majority pore size distribution and SEM for obtaining visual information regarding pore size and distribution. Other slurry based characterisations like Zeta potential optimisation, for obtaining stable non flocculated slurry, rheology study to obtain an estimate of proper flowability and thixotropic behaviour. Other mechanical characterisations include cold compressive strength and flexural bending to get an estimate of its strength values. The remaining portion of this thesis deals with the above mentioned parameters and procedures in an in-depth manner.

CHAPTER - 2

LITERATURE REVIEW

- Reference number written after the first reference number are additional referenced papers in conjunction with the main reference paper, the first number is indicative of the primary papers referred.

GELCASTING

Gel casting is an attractive ceramic forming process where we have the flexibility of casting and forming complex shapes [1]. Gel casting on the basis of fabrication route can be divided into two different types viz.

1. Aqueous route
2. Non-aqueous route

Considering the aqueous route of Gel casting we primarily have the following components that play a pivotal role in the modus-operandi of the entire process. The respective components being:

1. Solvent (primarily inorganic solvents)
2. Dispersant (To maintain slurry stability) [2]
3. Solute (The master material that needs to be casted)
4. Monomer (The unit that forms the basic polymerising unit)[2]
5. Cross linker (That which serves as a basic link b/w individual polymeric unit to form the much needed network)[2]
6. Initiator (To begin the reaction)[2]
7. Catalyst (To accelerate the reaction)[2]

INSIGHT TO GEL CASTING

Primarily in gel casting through the aqueous process we make use of a solvent and solute combination, the solute is added to the solvent at a fixed ratio of loading called as “solid loading”[3]. However, prior to adding the solute we add a fixed amount of dispersant to obtain a well dispersed slurry[3] that doesn’t show settling tendency and hence ensures proper workability during casting.

Having added the dispersant we gradually add the solute with optimum stirring to obtain a proper deflocculated solution. This solution is now subjected to addition of the respective

monomer and cross linking units [4,1] that polymerise on the subsequent addition of initiators followed by catalyst[4,1].

ADVANTAGES OF GELCASTING OVER AVAILABLE CASTING METHODS

Gel casting certainly expresses abundant versatility over other casting methods as far as casting advantages are concerned few of them are sequentially enumerated below:

1. Extremely versatile fabrication technique which is not powder specific can go in with multiple varieties of materials which can go in as the primary material to be casted .
2. Extremely attractive method for the fabrication of complex shapes e.g. Turbine rotors etc. [4,1]
3. The gel casting process is effectively a quick process to undertake so saves time and simultaneously though its quick setting but still then provides sufficient time for the material to be worked with hence doesn't hamper material workability.
4. Gel casted bodies have decent strength values both in the green as well as fired state on being compared in their respective categories.
5. Molding and demolding is an easy affair.
6. Gel casted green bodies have excellent machinability, which provides them an upper hand as far as pre firing shaping is concerned [4].

REAGENTS PREFERRED

In due context of the above process we have the following precursor selection as referred from the literature:-

1. Dispersant:

Dispersant primarily enhances the zeta potential of the slurry system thereby providing effective shielding, and hence prevents agglomeration, however excess of dispersant again leads to overcrowding and hence reduction in zeta potential thus an optimum concentration of deflocculants must be used. pH also plays a pivotal role in dispersant action[5].

Examples- Sodium hexa Meta-phosphate, Darvan-C [5]

Dispersant Chosen: Darvan-C which happens to be composed of ammonium polymethylacrylate, can be used at varying alkaline pH ranges, burn out temperature initiates at 232 degree Celsius. This is completely soluble in water systems[5,21].

2. Monomer & Cross linker:

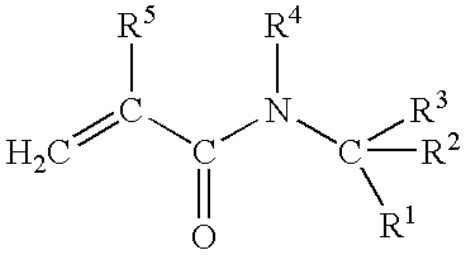
Monomer forms the framing unit and crosslinker forms the unit that links the monomers into one compact gelled entity giving them firmity and fixture and strength with a definite strength. Acrylamide systems come handy in the gel casting process .

Examples- Methacrylamide (MAM),

Poly (ethyleneglycol) 1000 dimethacrylate (PEG (1000) DMA) [5,13]

Monomer & Cross linker chosen: MAM/MBAM (methacrylamide/ methylbis-acrylamide) system was chosen.

(I)

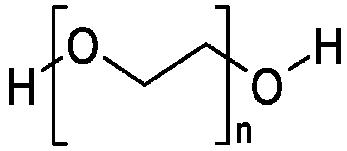


MAM schematic above works primarily on the basis of radical polymerisation as depicted by the N atom above in the schematic. Moreover present day MAM/MBAM systems present extremely low levels of toxicity when used for aqueous gel casting hence can be used [6].

3. De-exfoliant:

Surface oxidation is an imminent problem on account of exposure to the free atmosphere, hence inert atmosphere is needed , but the process being costly we make use of de-exfoliant which coats the top layer of the ceramic forming an inert layer over it thereby preventing scaling of the surface .

De-exfoliant chosen: PEG (600) polyethylene glycol was brought in as the de exfoliant.



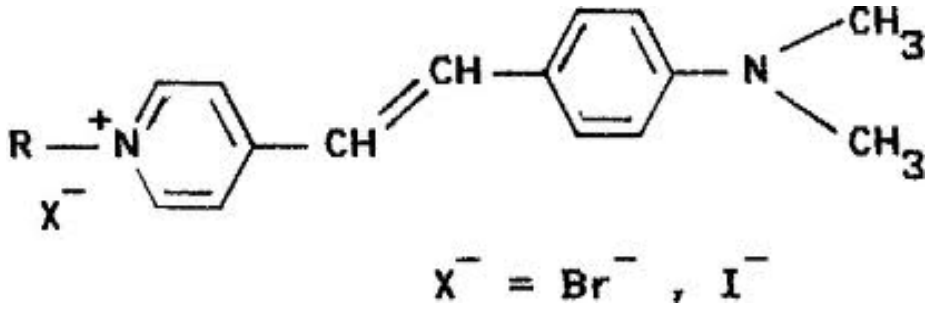
PEG schematic, which clearly illustrates oxygen bonding throughout.

4. Surfactants:

Surfactants are used to introduce bubbles into the cast thereby entrapping pores and hence increasing the body porosity. The essentially reduce the slurry surface tension as a consequence of which bubbles barge in to the surface and increase porosity, they stabilise the bubbles [6].

Examples: CTAB, ezee, triton x……..[7,18]

Surfactants chosen: The surfactant chosen is CTAB (Cetyl Trimethyl Ammonium Bromide)



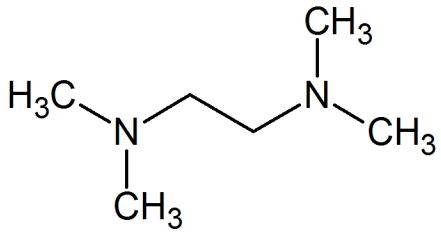
Schematic of a CTAB molecule.

5. Initiator & Catalyst: Initiator and catalyst as usual promote and accelerate the kinetics of the gelling process

Examples: APS (ammonium persulfate), AZAP (azobis (2-amidinopropane) HCl),
TEMED (Tetramethyl-ethylene-diamine) [7,8,1]

Initiator & Catalyst chosen: APS ($NH_4S_2O_8$), TEMED [8].

Temed is a very active catalyst and enhances the setting rate by manyfolds.



Schematic of TEMED.

Other materials used for the process of gelcasting that have received recent prominence are Natural Gelatine that can serve as a suitable gelling agent [9] replacing other toxic polymeric materials that have proved as necessary health hazards. This gelatine can be used to produce both dense as well as porous ceramics.

Another no toxic material that has set a trend apart is Agarose solution which is a complex carbohydrate [10] this has served as an excellent gelling material coupled with zirconia this has produced a mindblowing figure of 99% dense material.[10].

Reactive Alumina (α-Alumina) - The colourless hexagonal crystal form of alumina is else known as reactive or **α-Alumina.** This form is stable above 1200^0 Celsius prior to which we have a stable gamma phase of Alumina which is cubic in nature, as far as its crystal structure is concerned. With a molecular mass of 101.48 a.m.u this substance has a melting point of 2054^0 Celsius and a boiling point of 2980^0 Celsius and a density of 3.97g/cc.

The following properties are attributed to alumina

1. Excellent wear resistant properties.
2. Inert to a broad range of acid as well as alkali attack.
3. Has a very ideal and near perfect tendency to be casted and shaped.
4. Strength and stiffness values are notably on the higher side.
5. Has excellent tribochemical and tribomechanical behaviour.
6. Wonderful thermal spalling resistance and thermomechaniacl properties.

Porous alumina manufactured by gelcasting can be used a microporous membrane in specialised sectors like the food processing industry[1]. on account of its non leachability. Conventional filtering action for normal water and other corrosive liquids also form a part of the application. It can also be used for light weight frames and structurally insulating materials.

Using the above precursors a gradient porosity layer wise can be incorporated, this shows increased efficiency towards separation and reduced resistance towards filtration [11,12]. This is what we define by functional ceramics and here the function shall be porosity all through out. Their processing and post fabrication machinability are also excellent[12].

AIM OF THE PROJECT:

After studying the literature it has been found that different porosity gradient was a feasible endeavour using the organic route, hence the target accordingly was set as:-

To gel cast layers of porous alumina by surfactant action and then prepare multiple layers of different porosity and then seamlessly integrate them into one body which can serve in as a particulate filter material.

Other important stepwise building of the targets include:

1. To optimise zeta potential and rheology study of the slurry
2. To manufacture defect free porous gel casted body
3. To test the Apparent Porosity and Bulk Density of the fabricated body
4. To obtain pore size distribution by mercury porosimetry
5. To study its microstructure through SEM study.
6. Fabrication of a seamlessly integrated functional body where porosity is the function
7. To check its compressive and flexural strength.
8. Examining its practical applicability towards particulate filtration.

CHAPTER - 3

EXPERIMENTATION

PLANNING OF THE PROJECT EXPERIMENTATIONS

Primarily this project had its experimental procedures categorised into 4 discrete parts we can either way say that this project had 4 different course of action throughout, the details of the process along with their objectives and the experimental procedures following them have been enlisted below in due sequence.

4 parts that determine the course of action:

- **3.1 STABLE SLURRY PREPARATION:**

  OBJECTIVE: To prepare a stable alumina slurry, the stability of whose varies as a function of pH, solid loading and Zeta Potential.

  The stabilized slurry doesn't settle with proper consistency and flow ability.

  CHARACTERISATION:

  1. Zeta potential at various pH and dispersant concentration to determine the optimum potential.
  2. Rheology in the viscometer.

- **3.2 FABRICATION OF GELCASTED BODY & ACHIEVEMENT OF HIGHEST POROSITY:**

  OBJECTIVE: To fabricate porous bodies by gel casting using the above stabilized alumina slurry assisted by surfactant[7,8] and finds the combination to maximum porosity. Water being the solvent, a combination of **7:3, 1:1, 1:4** ***Solid:Water*** loading ratio was tried out at the optimized value of dispersant used i.e. Darvan-C and the amount of surfactant (CTAB) was varied and the porosity was measured.

  CHARACTERISATION:

  1. Apparent porosity and bulk density.
  2. Mercury porosimetry.
  3. SEM

* A landmark maximum porosity of 60% was achieved under normal laboratory conditions.

- **3.3 FABRICATION OF MULTILAYERED BODY WITH CHANNELED PORES THROUGH GELCASTING BY SURFACTANT ACTION AND ADDITIVE ADDITION:**

OBJECTIVE: To fabricate bodies with channeled pores so that they can find practical use by usage of surfactant.

At solid loading of 1:1 and 7:3 we manipulate the amount of surfactant over (2.5, 5, 7.5%) w/w and obtain multilayered ceramic with channeled pores & each layer having varied porosity.

* Surfactant addition showed positive results whereas additives didn't respond well.

MECHANICAL CHARACTERISATION:

1. Compressive Strength
2. Flexural Strength

- **3.4 FABRICATION OF A FUNCTIONALLY GRADED POROUS MATERIAL WITH LAYERS OF VARYING POROSITY FOR FILTER APPLICATIONS:**

OBJECTIVE: We seamlessly integrate a multilayer body with varying porosity and having channeled pores which can behave as filter. This can behave as a functionally graded material[11,12].

NOVELTY: We can control the porosity and pore dimension at our will unlike normal filters which are made by varying the packing b/w coarse medium and fine grains and are much more dependent on the material available at hand. In the process of gel casting we can bring about a desired change in the amount and distribution of porosity throughout the material. The variables for controlling porosity are far more flexible and easy to control in the above mentioned process of gel casting [11].

EXTENSION 3.1

Slurry stabilisation

3.1.1 ZETAPOTENTIAL OPTIMISATION

OBJECTIVE: To obtain a stable deflocculated slurry that can promote convenient workability by optimising parameters like pH, dispersant concentration, zeta potential and study the rheological properties of the slurry.

* We know as ascertained from previous papers stability of a slurry depends on its resistance to flocculation and hence the parameter that can parameterise the amount or magnitude of flocculation is given by "Zeta potential" [1] . Hence increased zeta potential means an increased shielding and hence reduced flocculation.

Zeta potential varies as a function of:

1. pH of the existing solution
2. Amount or concentration of the dispersant used i.e. Darvan-C [5].

* It doesn't depend on the amount or concentration of the material.

Below mentioned will be the steps in sequential arrangement one following the other as has been conducted in the respective experiment.

1. First a slurry of 7:3 solid loading was prepared by taking 30 ml of distilled water and to it we added 0.5%w/w(of solid loading) dispersant i.e. Darvan-C. Hence the amount of Darvan-C added was 0.35 grams.

   * We simultaneously prepare 2 other independent samples with dispersant concentration 1% w/w (of solid loading) and 1.5% w/w (of solid loading), hence the amount of dispersant added would be 0.7 grams and 1.15 grams respectively. We now have 3 independent solutions of water and dispersant.

2. To the above mentioned solution we allocate identifiable codes for easy identification and to every individual sample we add reactive alumina (our precursor material), gradually with optimum mixing. The amount of alumina added would be 70 grams in 30 grams of distilled water as specified by the solid loading ratio.
3. Now we have three independent slurry solutions with us.
4. In three independent clean beakers we take some amount of distilled water, the amount of water is not fixed any amount can be taken.

5. To these independent beakers we add a pinch of the respective slurries made and stored under the above identifiable codes.
6. Then after adding the desired slurries we ultrasonicate them for a 10 minutes interval till we don't get a completely non turbid clean and dispersed solution.
7. If the solution still shows turbidity and is not cleanly dispersed we redilute it again and again subject it to ultrasonication.
8. This step is carried till we don't obtain a clean and clear solution.
9. Then having obtained a clean solution from each of the individual beakers we empty the clean solution into five independent test tubes demarcated as pH(1, 3, normal, 9, 11).
10. The pH of the respective solutions as demarcated in the test tubes were taken to that value by the addition of either dilute Nitric acid (for acidic pH) or Ammonium Hydroxide (for basic pH).
11. The individual samples from the test tubes were taken and their zeta potential was measured in the zeta potentiometer.
12. The pH against zeta potential at different dispersant concentration was noted down.
13. The previously made alumina slurry was kept standing for 24 hours to check their settlement.

POINTS TO STAY CAREFUL ABOUT

1. During this entire ordeal we must be careful about the exactness of the amounts of reagent being taken we must try and maintain their exactness.
2. During ultrasonication we must ensure that the solution is aptly dispersed by holding it against light else a redilution is a must.
3. During Zeta Potential measurement we must make sure that the amount of material going into the case must not allow any bubbles into it, it shall give a faulty reading.
4. Bubbles can be removed by appropriately syringing out the air form the syringe before injecting into the case.
5. Stay careful of the acids and bases used Nitric acid can attack skin forming Xanthoproteins that turn skin colour yellow, ammonium hydroxide can be nauseatic.

3.1.2 RHEOLOGY STUDY

OBJECTIVE: To study the rheological behaviour of the prepared slurry and make a comparative analysis of various solid loading considered in the experiment.

The steps envisaged in the given trial are the following:

1. Three distinct slurries were prepared with solid loading (alumina:water) as 7:3, 8:3, 9:3.
2. The amount of dispersant used as optimised by the previous experiment equated to 0.5% w/w of the solid loading that is 0.35 grams, 0.40 grams and 0.45 grams respectively.
3. The method of preparing the slurry was identically the same as mentioned in the previous page.
4. The slurry was then individually categorised under three different sections for the three different levels of solid loading.
5. The three different demarcated slurries were then put to test in a rheometer.
6. The rheometer operating at a fixed rpm for a desired amount of time that is programmed checks for the slurry viscosity along with variations in the shear rate and shear stress data.

POINTS TO STAY CAREFUL ABOUT

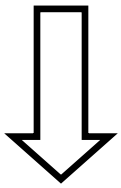
1. During this entire ordeal we must be careful about the exactness of the amounts of reagent being taken we must try and maintain their exactness.
2. Avoid body contact or any other contact with the viscometer as it might result in the production of erroneous data by taking up the external vibration induced by these objects.

EXTENSION-3.2

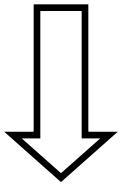
Fabrication of gel casted body

We have the following fabrication steps for gel casting:

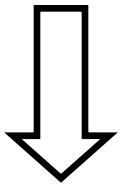
Take Powder (Reactive Alumina) and according to the loading ratio take the amount of water (distilled).



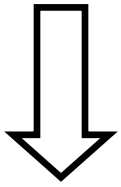
First to the water add 0.5% w/w dispersant and allow it to slowly stir and then gradually add alumina to it.



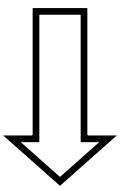
To it add monomer & crosslinker (MAM +MBAM) total 15% by weight divided in the ratio 3:1.



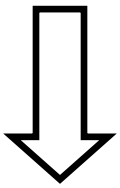
To it add de exfoliant (3% w/w PEG 400) and followed by surfactant for pore entrapment. Surfactant mass may vary depending on the amount of surfactant (CTAB) added which has been varied over the range (2.5%, 5%, 7.5%) w/w of the solid loading.



Adequate stirring followed by addition of initiator (APS) and a pinch of catalyst (TEMED) is added and is casted.



Room temperature drying for 24 hrs followed by controlled drying at 50^0 Celsius for 5 hrs.



FIRING at 1600^0 Celsius

POINTS TO STAY CAREFUL ABOUT

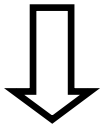
1. MAM and MBAM species should not be allowed to stand for a long time as they start self polymerisation.
2. The catalyst TEMED is increasingly corrosive and hence it must not be brought in physical contact with the human body.
3. TEMED results in rapid hardening of the cast hence after addition of catalyst the casting should be done as soon as possible.
4. Mixing should be proper at the end of every step to ensure proper homogeneous mixing.

EXTENSION 3.3 & 3.4 (combined)

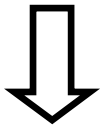
Fabrication of a functionally graded porous material.

In this section we have tried to manufacture individual layers with varying levels of porosity and their properties have been studied. Properties primarily relate to their mechanical properties viz: Cold crushing strength and Flexural Strength. Having obtained a fair impression about their strength values we tried to seamlessly manufacture a filter body comprising of individual layers of porous gel casted alumina which have been seamlessly integrated by the below mentioned process.

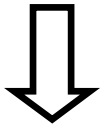
We go for individual castings of monolayer bodies one over the other.



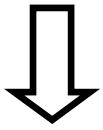
First cast one layer with certain porosity and allow it to dry.



Then cast a layer on top of it with increased porosity and follow the same process for further castings.



Drying is extremely critical first has a 24 hrs air drying followed by 5 hrs at 50^0 Celsius.



Firing schedule is very delicate heating rate should be < 1^0/min, fired at 1600^0 Celsius.

PROPERTY ASSAY

1. APPARENT POROSITY & BULK DENSITY: We take an assay of the formation of open pores; this gives us an impression of the amount of porosity in the body which is an important parameter of study in this project. Porosity primarily envisages three important parameters that determine it viz;

Dry weight (DW) - Weight of the normal sample

Soaked Weight (SW) - Weight of the sample after being soaked in the desired liquid (distilled water here), primarily refers to amount of water that has percolated into the pores.

Suspended Weight (SuW) - Refers to the weight of the sample minus the buoyant force of the fluid acting on it.

APPARENT POROSITY = (SW - DW) / (SW - SuW) * 100%

Similarly bulk density is inversely proportional to apparent porosity and gives the amount of material in the bulk minus the porosity given by

BULK DENSITY = DW / (SW - SuW) g/cc.

2. ZETA POTENTIAL: Measured to give the amount of slurry stability, primarily parameterises slurry stability with a potential called as Zeta Potential, measured by a zeta potentiometer. Higher the zeta potential more is the shielding. It is detected in millivolts.

3. VISCOSITY & RHEOLOGY: Slurry viscosity and rheology was measured using the rheometer, a comparative assessment of shear stress vs. shear rate and viscosity with time was given for different solid loadings as mentioned above in the experimentation column.

4. MERCURY POROSIMETRY: Works on the principle of intrusion and extrusion of mercury, substantially detects the amount of porosity and majority pore size distribution. This is made possible by injecting mercury into the pore more the volume of intrusion of

mercury into the pores more is the density of the particular pore having a definite size. Porosity trends through porosimetry studies have been shown by changing surfactant concentration along with solid loading.

5. MECHANICAL PROPERTIES: The faculty envisaging mechanical properties comprised of:

Cold Crushing Strength (CCS) - interprets the maximum compressive strength bearing capacity of the body. The ratio of length:depth ratio was maintained b/w (1-1.5) of the respective samples. CCS of single layered and seamlessly integrated bi-layered bodies have been measured by varying surfactant concentration as well as solid loading. This is to primarily infer their load taking behaviour under the application of compressive load when made to behave as a filter material.

Flexural Strength - Measures the tensile strength of the body. It is governed by the formula: $(1.5 * P * l) / b * d^2$, where

L = sample length, P = Load on the sample, b = breadth of the sample and d = depth of the sample.

According to ASTM standard (C-1684) sample length should lie b/w (25-85mm) and diameter b/w (1-8mm).

6. MICROSCOPY ANALYSIS: Two distinct types of microscopy analysis were performed viz:

1. SEM: Was performed to obtain a visual impression of the pore size and porosity distribution across a sample.

CHAPTER - 4

RESULTS, ANALYSIS AND DISCUSSION

4.1ZETA POTENTIAL ANALYSIS

The zeta potential was measured by the previously mentioned steps in the experimental section of this thesis. The data has been tabulated below and an appropriate reasoning to the given behaviour was given.

An initial settlement test was done with a relatively used and older variety of Darvan-C. The amount of the dispersant was varied for a fixed solid loading of 7:3. The slurry was divided into five different sections demarcated as (0.5%, 0.7%, 1.0%, 1.2%, 1.5%) based on the amount of dispersant added and they were kept standing for 24 hours and their settlement behaviour was checked.

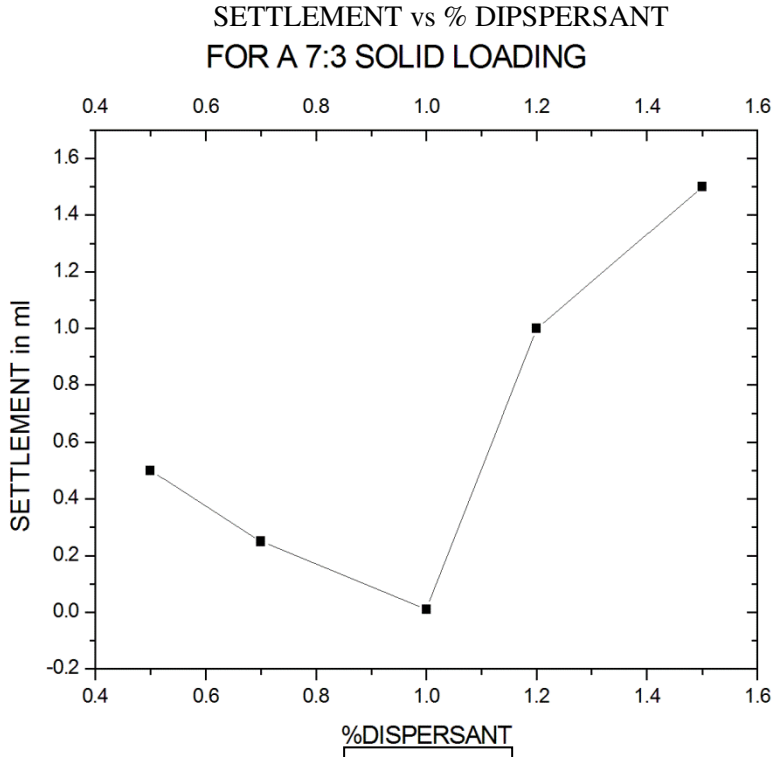


Fig 4.1.1

As evident at a percentage of 1.0% of the total solid loading the amount of settlement was the least, hence as evident **(fig 4.1.1)** this must indicate the fact that we can have maximum dispersed slurry at 1.0% dispersant concentration. However we need to still a concrete evidence for the above mentioned assumption and hence we move towards the zeta potential test. Moreover the above mentioned Darvan-C sample was relatively old and hence was suspected to have shown anomalous properties, so a proper testing was essential.

TABLE 4.1

% DISPERSANT	pH	Zeta Potential (mV)
0.5	1.00	10.2
	3.00	8.73
	6.3	-37
	9.2	-66.1
	11.2	-8.98
1	1.25	0.5
	2.76	-0.221
	6.9	-21.7
	9.2	-51
	11.7	-27.9
1.5	0.98	-0.741
	3.05	2.67
	7.95	-0.475
	9.4	-35.9
	11.81	-11.3

ZETAPOTENTIAL ASSAY

From the above table**(4.1)** it is evidently clear that the amount of dispersant added that needs to stabilise the slurry is 0.5% of the solid loading as the zeta potential trend exhibited by the 0.5% dispersant concentration slurry has been uniform as depicted by the graph 4.2.2. The Zeta potential obtained has been maximised at -66.1mV at pH 9.2 which is certainly a promising and a full proof result. This value of zeta potential ensures maximum shielding and hence prevents alumina particles from agglomerating thereby providing better workability for the slurry. The 1% concentration dispersant also shows a decent shielding at -51mV; however we get an optimised value at a lesser amount of Darvan-C thereby reducing the organic load which is but certainly desirable. A pH close to 9.2 is automatically achieved when 0.5% (w/w) dispersant is added hence giving scope for the best shielding.

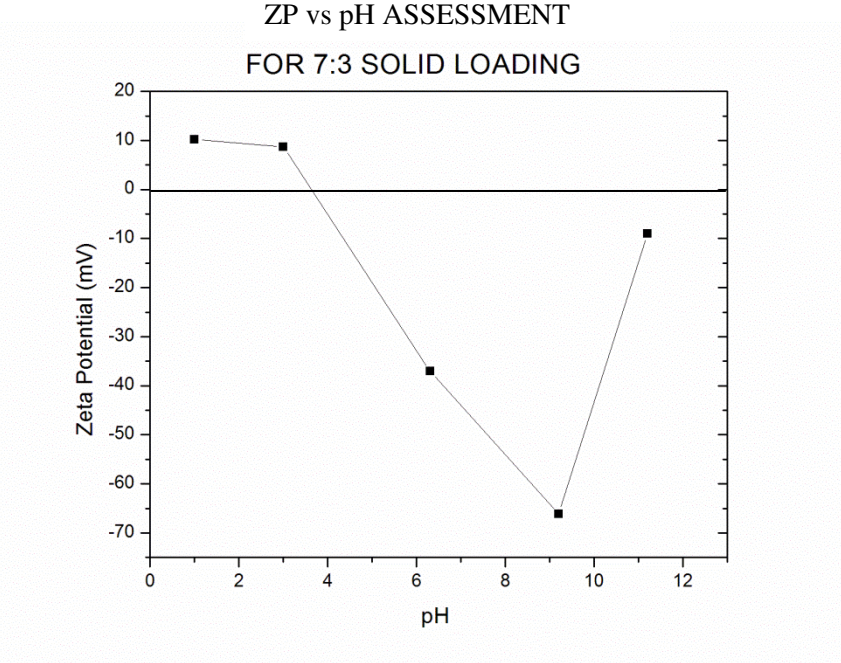


FIG 4.1.2

From the above curve we can evidently see the minima representing the maximum Zeta potential (only as far as magnitude is concerned), we also observe a single isoelectric point at pH 3.6 in **fig 4.1.2** however that certainly cannot be claimed of pure reactive alumina on account of dispersant (Darvan-C addition). The minimum zeta potential value of -66.1 mV could be ascertained from the above graph. The point of zero zeta potential is called isoelectric point.

4.2 RHEOLOGY

Typical rheological behaviour of the slurry was studied at different levels of solid loading to get an impression of the slurry flowability and conveniency during casting. Under the mentioned circumstances increasing the solid loading shall certainly hike the viscosity leading towards non flowability and hence casting problems. Hence to get a comparative evaluation of the amount of increase in the viscosity and variation in parameters like shear stress and shear rate testing were done for 8:3 solid loading and 9:3 solid loading along with 7:3 solid loading.

TABLE 4.2

	Time (s)	Viscosity (Pa.s)	Shear Rate (1/s)	Shear Stress (Pa)	Torque (mNm)
8:3 SOLID LOADING	1	0.0456	1.00E+03	45.6	7.73
	2	0.0452	1.00E+03	45.2	7.66
	3	0.0448	1.00E+03	44.8	7.59
	4	0.0444	1.00E+03	44.4	7.53
	5	0.0441	1.00E+03	44.1	7.47
	6	0.0437	1.00E+03	43.7	7.41
	7	0.0435	1.00E+03	43.5	7.37
	8	0.0433	1.00E+03	43.3	7.33
	9	0.0432	1.00E+03	43.2	7.31
	10	0.0431	1.00E+03	43.1	7.3
	11	0.0430	1.00E+03	43	7.29
	12	0.0430	1.00E+03	43	7.29
	13	0.0430	1.00E+03	43	7.29
	14	0.0430	1.00E+03	43	7.29
	15	0.0430	1.00E+03	43	7.29
	16	0.0430	1.00E+03	43	7.29
	17	0.0430	1.00E+03	43	7.28
	18	0.0430	1.00E+03	43	7.28
	19	0.0429	1.00E+03	42.9	7.28
	Time (s)	**Viscosity (Pa.s)**	**Shear Rate (1/s)**	**Shear Stress (Pa)**	**Torque (mNm)**

	1	0.0992	1.00E+03	99.2	16.8
	2	0.098	1.00E+03	98	16.6
	3	0.0968	1.00E+03	96.8	16.4
	4	0.0957	1.00E+03	95.7	16.2
	5	0.0946	1.00E+03	94.6	16
	6	0.0937	1.00E+03	93.7	15.9
	7	0.0928	1.00E+03	92.8	15.7
	8	0.0919	1.00E+03	91.9	15.6
9:3 SOLID	9	0.0911	1.00E+03	91.1	15.4
LOADING	10	0.0904	1.00E+03	90.4	15.3
	11	0.0897	1.00E+03	89.7	15.2
	12	0.0891	1.00E+03	89.1	15.1
	13	0.0885	1.00E+03	88.5	15
	14	0.088	1.00E+03	88	14.9
	15	0.0876	1.00E+03	87.6	14.9
	16	0.0872	1.00E+03	87.2	14.8
	17	0.0867	1.00E+03	86.7	14.7
	18	0.0863	1.00E+03	86.3	14.6
	19	0.0858	1.00E+03	85.8	14.6

	Time (s)	Viscosity (Pa.s)	Shear Rate (1/s)	Shear Stress (Pa)	Torque (mNm)
	1	0.0332	1.00E+03	33.2	5.64
	2	0.0326	1.00E+03	32.6	5.52
	3	0.0324	1.00E+03	32.4	5.49
	4	0.0323	1.00E+03	32.3	5.47
	5	0.0322	1.00E+03	32.2	5.46
	6	0.0323	1.00E+03	32.3	5.47
	7	0.0322	1.00E+03	32.2	5.46
	8	0.0322	1.00E+03	32.2	5.46
7:3 SOLID	9	0.0322	1.00E+03	32.2	5.47
	10	0.0322	1.00E+03	32.2	5.46

LOADING	11	0.0321	1.00E+03	32.1	5.43
	12	0.0321	1.00E+03	32.1	5.44
	13	0.0322	1.00E+03	32.2	5.45
	14	0.0322	1.00E+03	32.2	5.46
	15	0.0322	1.00E+03	32.2	5.45
	16	0.0322	1.00E+03	32.2	5.45
	17	0.032	1.00E+03	32	5.42
	18	0.0321	1.00E+03	32.1	5.44
	19	0.032	1.00E+03	32	5.42

RHEOLOGY ASSAY

From the above table**(4.2)** we have the following inference:

1. The viscosity values continually increase infact increase by a factor of 2 on taking the solid loading ratio from 8:3 to 9:3 as in **fig 4.1.6, 4.1.7**.
2. The shear rate values have been kept constant at 10001/s.
3. Viscosity decreases with increasing time keeping rest other parameters same hence we can certainly infer that viscosity is time dependant here and the slurry "Thixotropic".
4. The trend of variation of shear rate, viscosity and time has been elucidated in the graphs**(4.1.4,4.1.5)** following this.

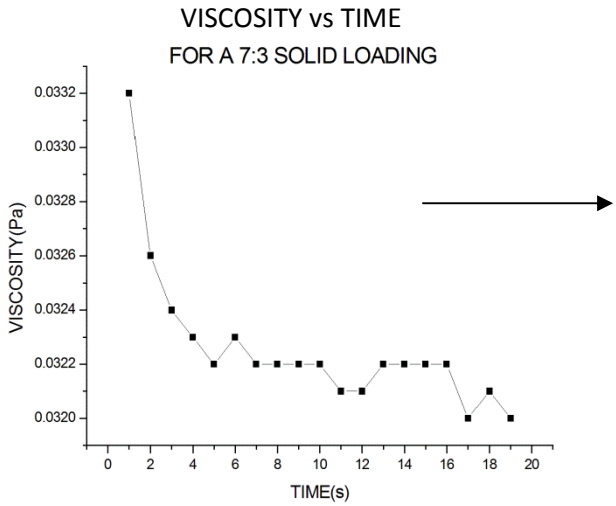


FIG 4.1.3

In **fig(4.1.3)**The solid loading that we shall be preferring , has the least viscosity in comparison to the other loadings on account of reduced solid loading, however slurry behaviour though thixotropic yet viscosity variation bit erratic after **5 secs** beyond 0**.322 Pa**. However this doesn't pose any serious problem as the overall slope of the graph reduces downwards proving its thixotropicity.

VISCOSITY vs SHEAR STRESS

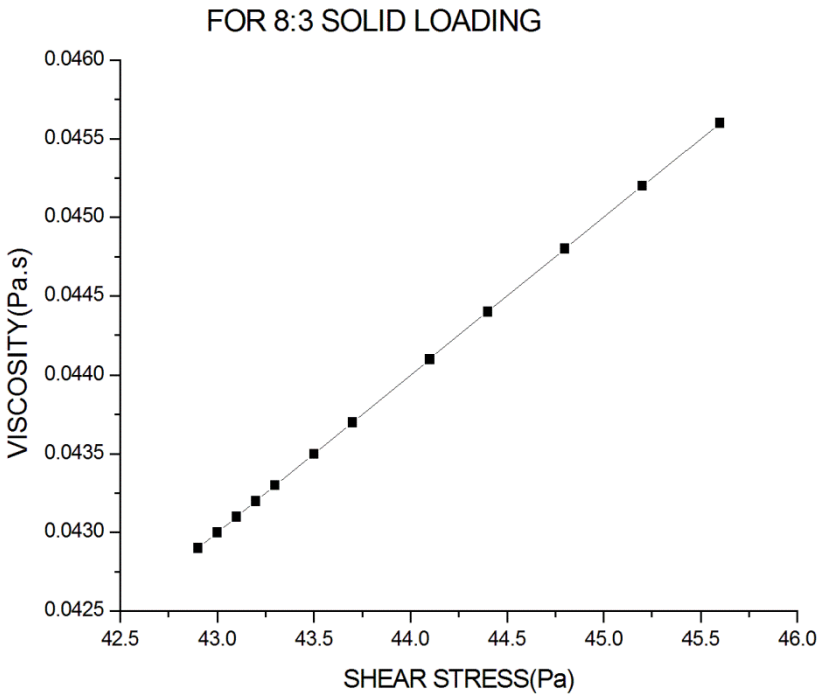


FIG 4.1.4

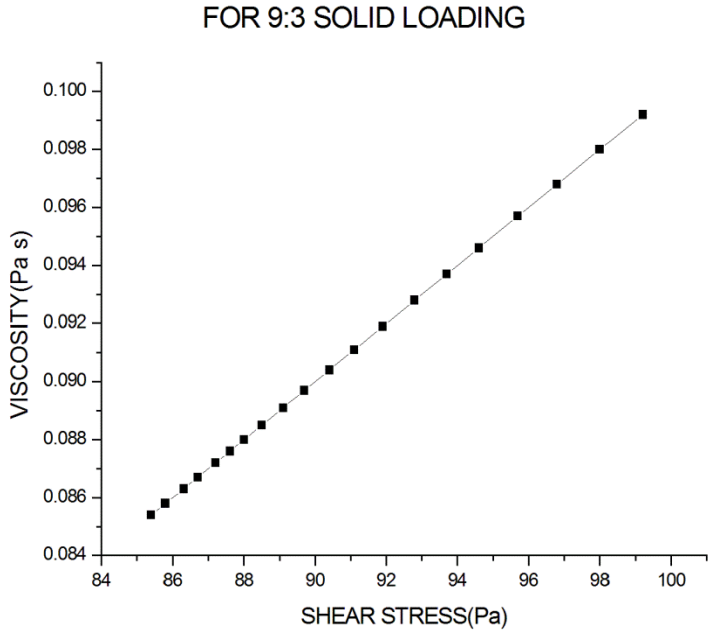


FIG 4.1.5

Above figures depict a linear relationship of shear stress with viscosity **fig(4.1.4,4.1.5)** which is but obvious at a constant shear rate when stress by underlying layer increases the reluctance or inertia to flow hence viscosity increases. The slope being the shear stress as mentioned equal 10001/s. Hence viscosity confirms the equation, Shear Stress=-viscosity*(Shear Rate)^n

Depending on the value of n we shall decide the slurry rheology

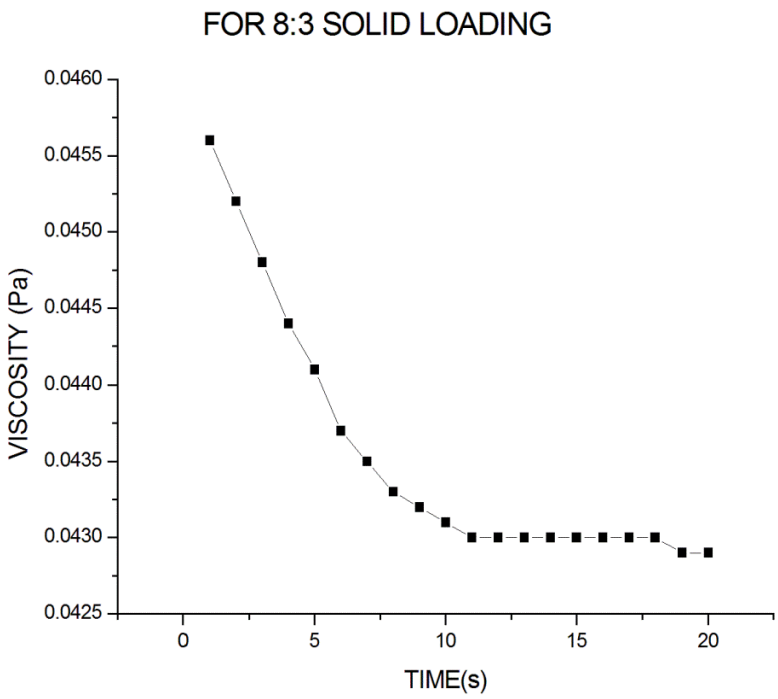


FIG 4.1.6

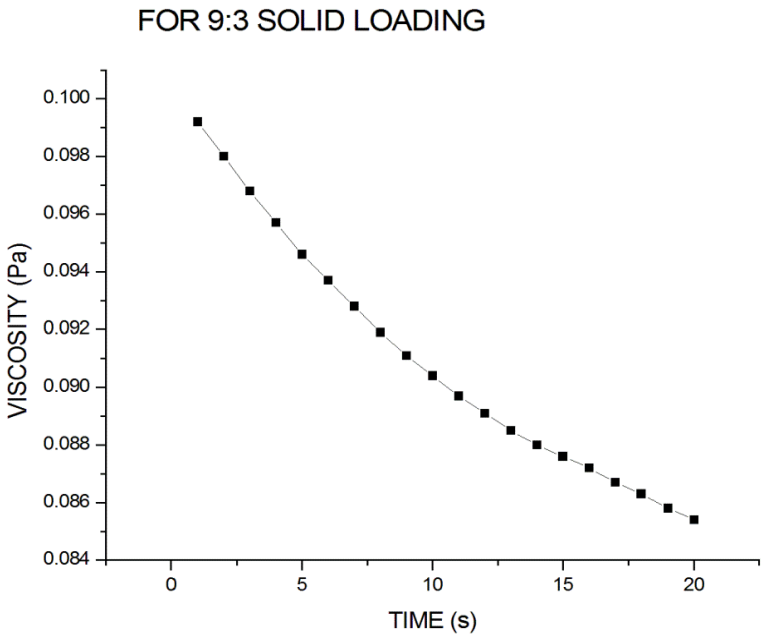


FIG 4.1.7

From the above plot of viscosity **fig(4.1.5,4.1.6)** vs time its inferred that viscosity reduces with increaing time. The first 20 seconds of the test has been plotted. The above two curves have been compared between 8:3 solid loading and 9:3 counterpart of its, however 9:3 solid loading shows a gradual fall in the viscosity value compared to 8:3 which falls steep suddenly between 7 to 10 seconds. This prompts that 9:3 slurry presents a better rheological property except for its increased viscosity value(4 times 8:3). Thus 9:3 slurry could be preferred for

our casting purpose but given to the fact that we need a porous body an increased soid loading might hinder the same hence finally 7:3 solid loading was preferred and in an attempt to further increase porosity solid loading in subsequent analysis has been reduced to 1:1.

The below mentioned curve shows a comparative assay of the viscosity behaviour of (7:3, 8:3, 9:3) slurry with time .

fig 4.1.8

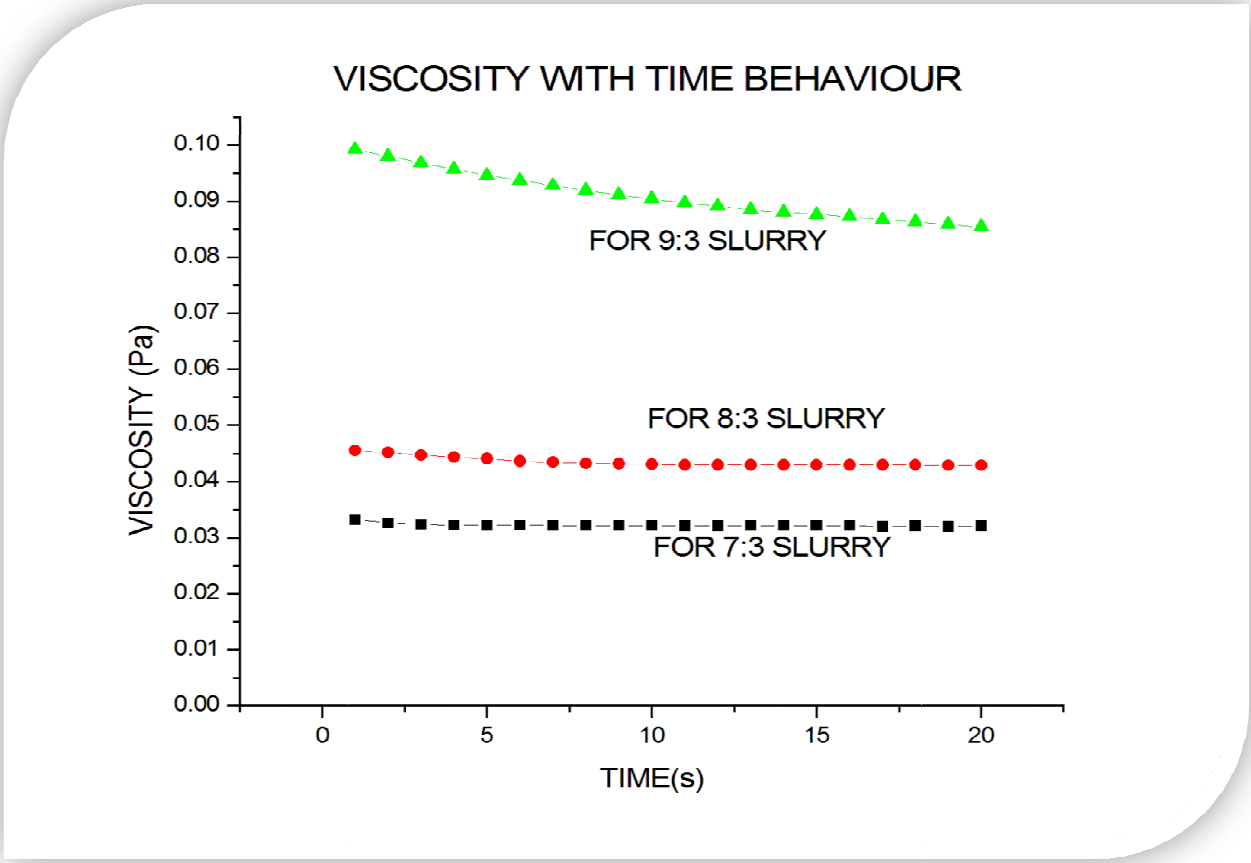


FIG 4.1.20

In fig 4.1.8 we observe that the thixotropic behaviour for 9:3 solid loading is the best and the most uniform in its category however on grounds of porosity reasons it had to be done away with moreover we used a 1:1 slurry for further increment in porosity values which showed us promising results which we shall unfurl in the due course of this thesis.

4.3 APPARENT POROSITY & BULK DENSITY

Apparent porosity and bulk density of the respective gelcasted bodies were tested . The solvent used to percolate the pores was distilled water, however other solvents like kerosene could also be used. The procedural formulae for testing the desired parameters has been given in the characterisation column.

However during testing certain care must be taken while measuring the soaked weight (Sw) and the suspended weight (Suw). During measurement of suspended weight we need to be careful about the fact that while hanging or suspending the material with the help of a hanger into the solution, no part of the beaker must come in contact with the hanger or the material else that would result in erroneus data.

Moreover during the determination of soaked weight we need to patch the drenched body with a wet cloth or wet tissue paper to remove the surface water, else on account of capillary action we might witness a suction of water from the pores, which certainly is undesirable.

AP & BD have been segmented into two primary types:

1. AP & BD for monolayer casting (1:1, 7:3 solid loading) - We have a single layer casting, casted at 1:1 and 7:3 solid loading. Their surfactant concentration has been varied over three range viz: 2.5%, 5%, 7.5% by weight of solid loading and their respective AP & BD's have been measured.

2. AP & BD for bilayer casting (1:1, 7:3 solid loading) - Here quite akin to the above made paragraph we have seamlessly integrated two layers.Each seamlessly integrated body has been manufactured at a particular solid loading, and we have tried out two different solid loading viz: 1:1 and 7:3. Now for every single body we have had bilayer castings where two layers are casted one upon the other with the topmost layer having the highest surfactant concentration. The coding for the above bodies has been 2.5-5, 5-7.5, this signifies that for 2.5-5 the bi-layered body has the upper layer casted at 5% surfactant concentration and the lower at 2.5%. This applies to other codings too. The reason behind testing for bilayered bodies was to determine the property of the integrated bodies i.e. do they lie between two extremeties of the property of individual layers or show some different trends.

The following table illustrates the data:

TABLE 4.3

CODE	AP (%)	+/- ERROR	BD (g/cc)	+/- ERROR
MONOLAYERED				
7:3 (2.5%)	20.68	0.64	3.16	0.02
7:3 (5.0%)	24.02	0.62	2.97	0.1
7:3 (7.5%)	27.65	1.45	2.88	0.05
1:1 (2.5%)	35.11	2.1	2.8	0.07
1:1 (5.0%)	57.2	2.0	2.67	0.2
1:1 (7.5%)	45.0	2.2	2.67	0.04
BILAYERED				
7:3 (2.5-5%)	27.0	1.10	2.83	0.1
7:3 (5.0-7.5%)	28.11	2.2	2.8	0.19
1:1 (2.5-5%)	41.2	2.23	2.46	0.11
1:1 (5.0-7.5%)	47.2	1.9	2.30	0.07

AP & BD ASSAY

We can certainly infer the following from **Table 4.3**:

1. Apparent porosity and Bulk density show a regular trend with increase in surfactant concentration and reduction in solid loading AP increases and BD reduces as shown by the graphs following this page.
2. The highest porosity attained was 59.2 as shown by the notch point (**fig 4.1.9**) and its average corresponding data was 57.2 as plotted however the respective BD(**fig4.1.10)** values showed a greater difference one being 2.232 g/cc and the other being 2.67 g/cc. The only anomality in the entire assessement.
3. The maximum registered error margin lies b/w 4-5% on an average we have error margins of 2-3%.
4. The properties of the multilayered (bilayered) **fig 4.1.11, fig4.1.12** bodies showed intermediate results between the extremeties as but expected.
5. Below mentioned we have a graphical assessement of the same.

FOR A SINGLE LAYER CAST:

AP & BD ASSAY FOR A SINGLE LAYER CAST

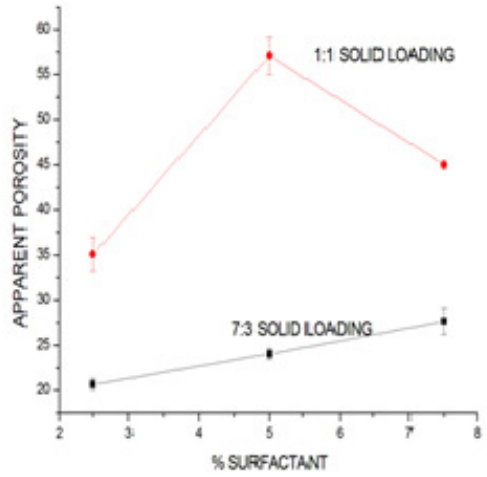


FIG 4.1.9

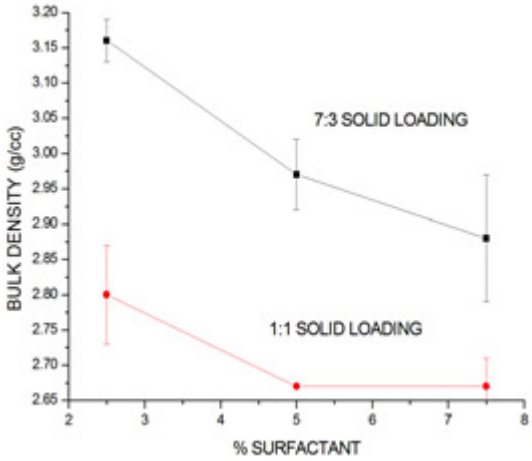


FIG 4.1.10

FOR A DOUBLE LAYER CAST:

AP & BD ASSAY FOR A DOUBLE LAYER CAST

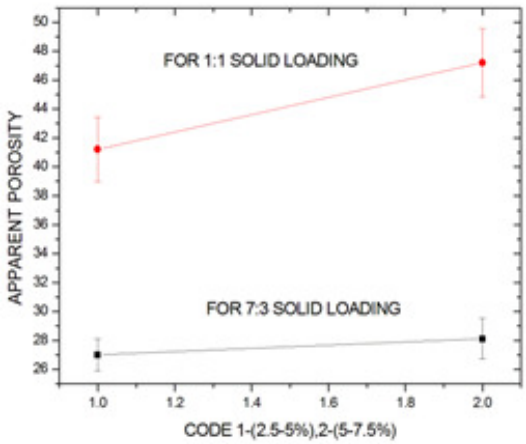


FIG 4.1.11

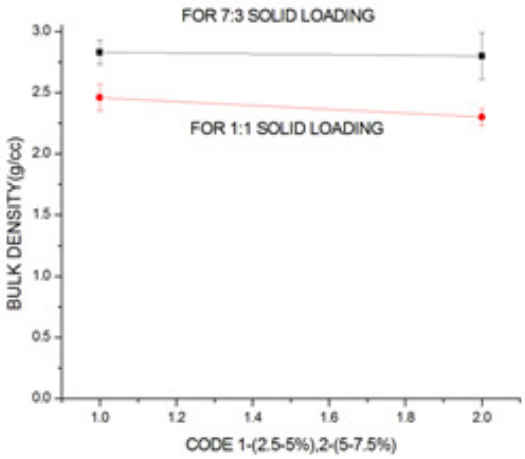


FIG 4.1.12

***CODE in the X axis for fig 4.1.10 and 4.1.11 imply 1 for 2.5-5% bilayer casting and 2 for 5-7.5% bilayer casting, this coding is also used in future graphs and the allotment logic is the same**

4.4MERCURY POROSIMETRY:

Works on the intrusion and extrusion of mercury in and out of the pores. Increased volume of pores shall certainly mean more amount of intrusion and hence extrusion too. Hence in this specific experiment using the above principle we shall be measuring the majority distribution of pore sizes in the body and if at all its suitable for filter applications other variations of pore sizes with change in solid loading and surfactant can also be ascertained about.

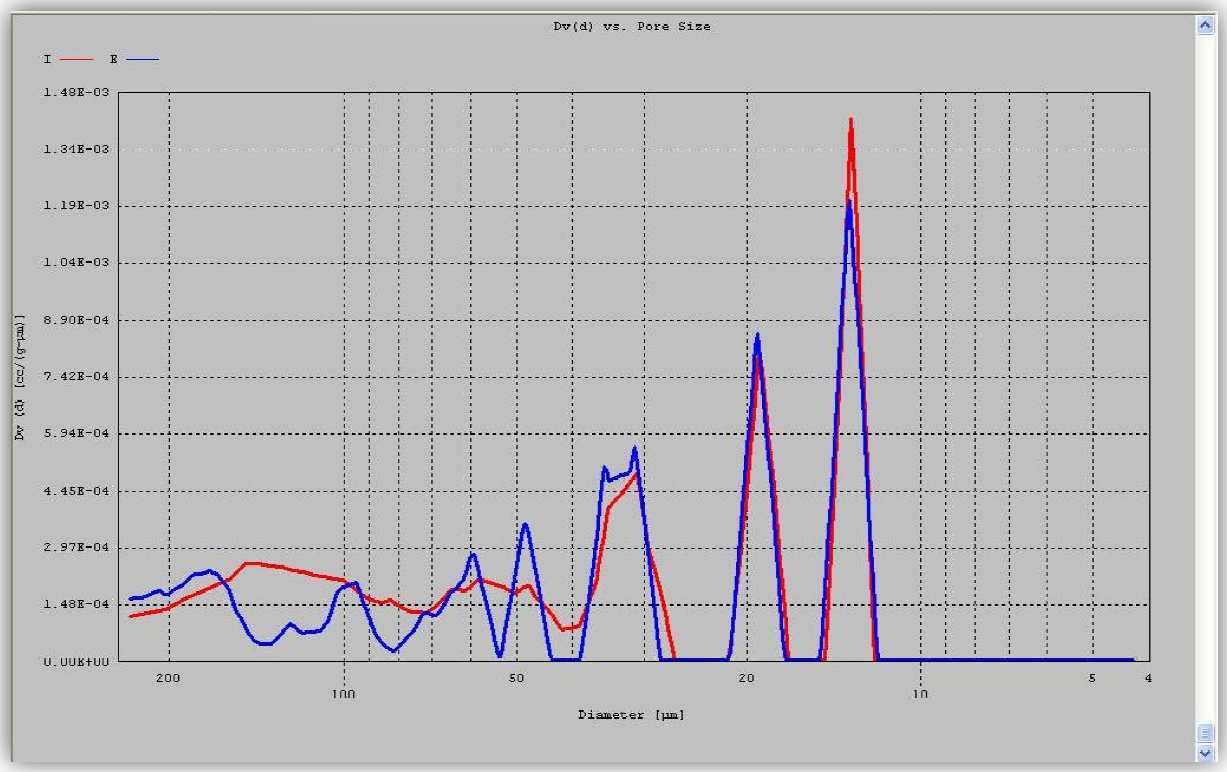


FIG 4.1.13

Porosimetry data of **7:3** sample with **2.5%** of surfactant concentration. As observed in fig **4.1.13** we can see the maximum concentration of pore size is around **15 microns.** The data shows a red and blue line that is representative of the intrusion and extrusion volume and the extrusion volume is indicative of the porosity distribution. Some amount of hysteresis or loss at a particular size is always there on account of incomplete removal of mercury from the pore of the same size, that amount of mercury is ejected from a pore of a different size. However no amount of mercury stays back in the body.

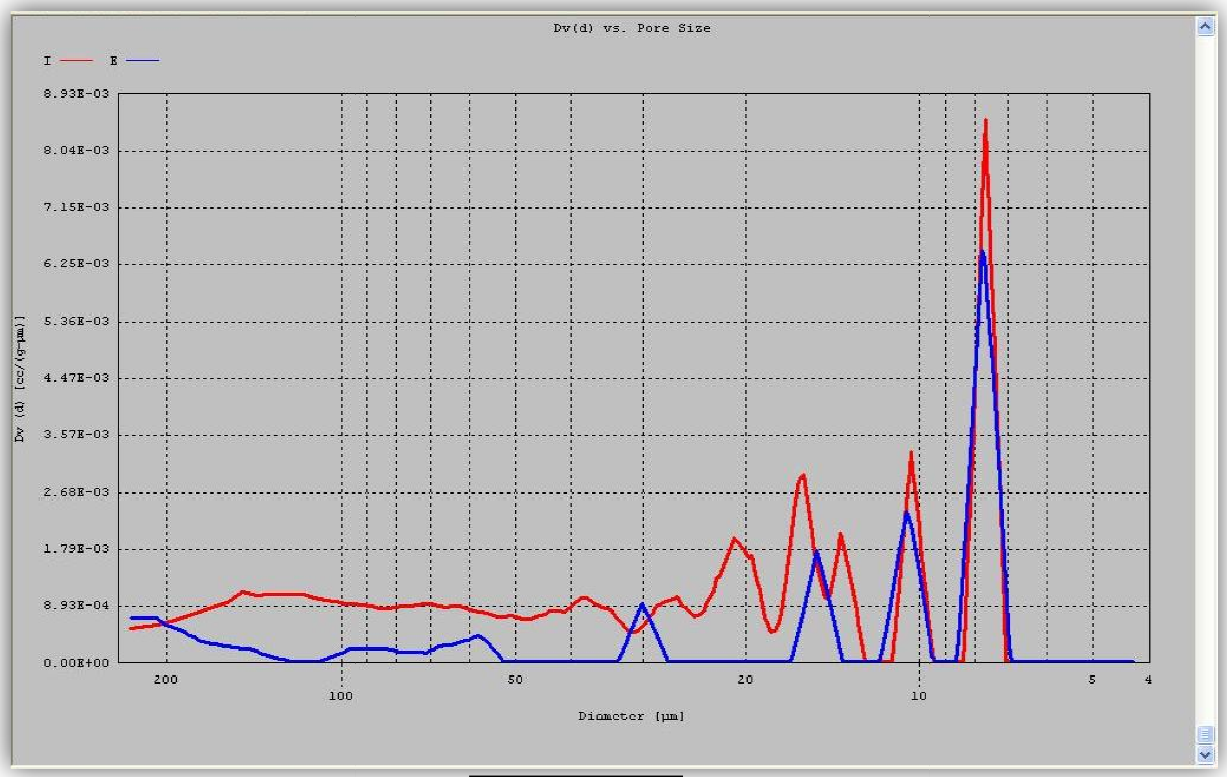


FIG 4.1.14

Porosimetry data of **7:3** sample with **5%** of surfactant concentration. As observed in fig **4.1.14** we can see the maximum concentration of pore size is around **7.5-10 microns.** The data shows a red and blue line that is representative of the intrusion and extrusion volume and the extrusion volume is indicative of the porosity distribution.

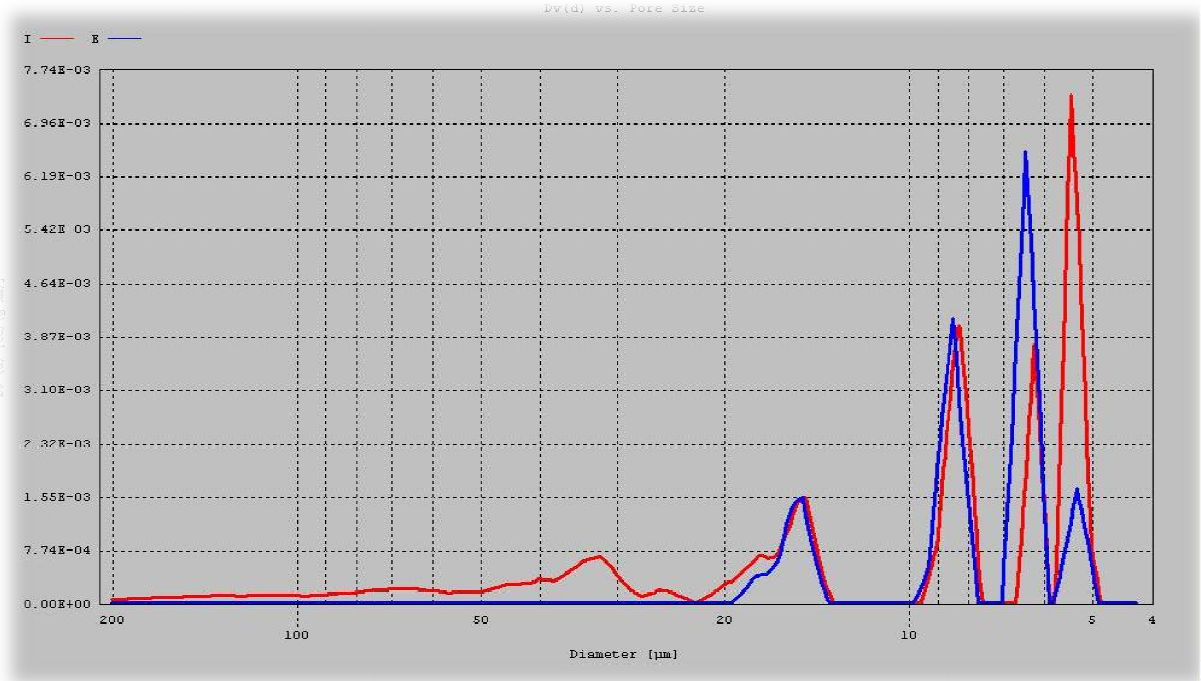


FIG 4.1.15

Porosimetry data of **1:1** sample with **5%** of surfactant concentration **fig 4.1.15**. As observed we can see the maximum concentration of pore size is around **5-7.5 microns.** The data shows a red and blue line that is representative of the intrusion and extrusion volume and the extrusion volume is indicative of the porosity distribution. **This is restrictive to many particulate impurities and is fit for being a microporous membrane.**

- From the above graphs its but evidently clear that pore size reduces with addition of more amount of surfactant, may be because of the increased entrapment of bubbles which finally coaslesce and burst to even finer units on exceeding their own surface tension.
- However its ascertained that a uniform gradation with repect to solid loading and percentage variation from top to bottom of a cast can certainly give us a variation in the pore size which when decreasing from top to bottom can give us handsome filtration results.

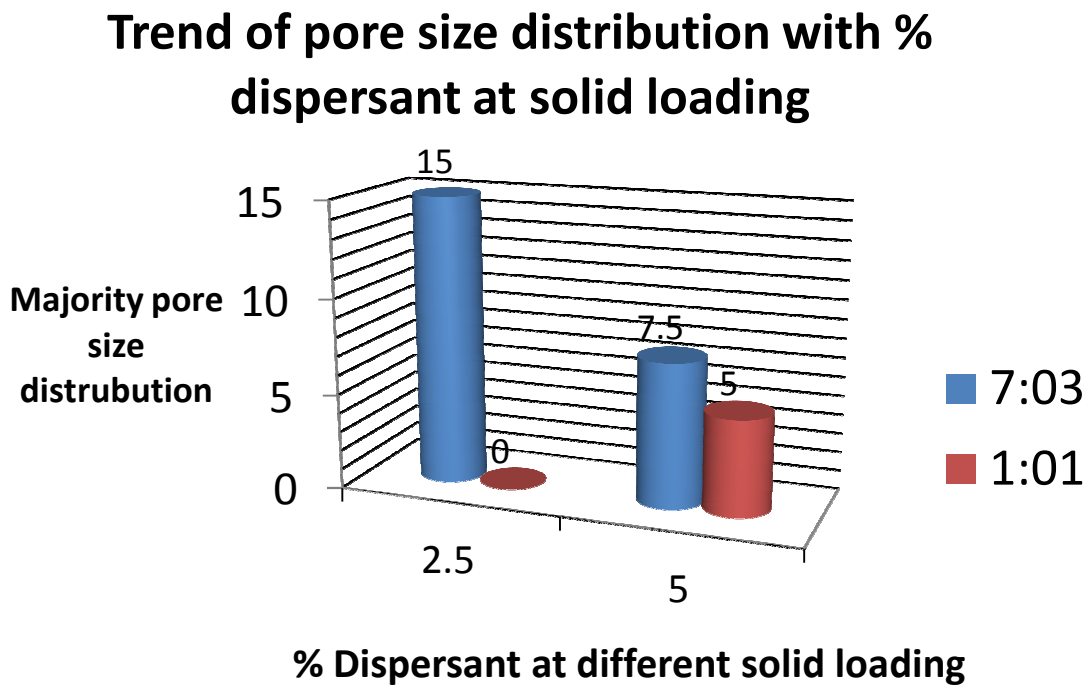


FIG 4.1.16

In fig **4.1.16** we have a comparative layoff of the three porosimetry data put together in the above graph which but evidently portrays the fact that with increasing surfactant concentration the pore size in majority reduces, the reason to which has been compiled above.

4.5COLD CRUSHING STRENGTH:

Filter materials are often subjected to stresses in the form of fluxes of incoming material as well as we also have the weight of the residual filtrate that piles up on filtering membrane. CCS values(**table 4.4**) were also computed on the same basis by changing surfactant concentration at a fixed solid loading and the comparison was made.

TABLE - 4.4

CODE	2.5 (7:3)	5 (7:3)	7.5 (7:3)	2.5-5 (7:3)	5-7.5 (7:3)	2.5 (1:1)	5 (1:1)	7.5 (1:1)	2.5-5 (1:1)	5-7.5 (1:1)	0 (1:1)
CCS (Mpa)	41.73	20.17	8.07	19.20	17.30	20	15.04	3.00	4.74	4.42	48.96

CCS ASSAY

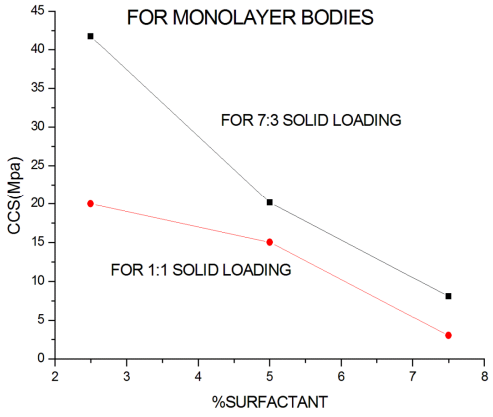


FIG 4.1.17

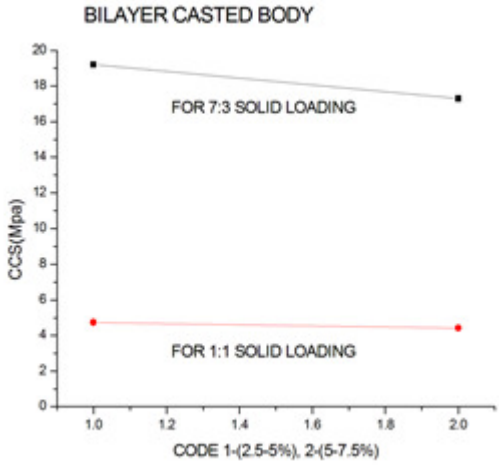


FIG 4.1.18

1. The CCS values(averaged) have reduced with increasing porosity, suggestive of the fact that strength reduces with porosity as per fig **4.1.17**.
2. There has been a considerable deviation from the ideal strength of dense alumina.
3. However strength values of bi-layered body fig **4.1.18** are intermediate b/w extreme configurations of mono layered porous bodies.
4. The amount of deviation however cannot be computed with ccs however a slight increase in porosity drastically reduces the **CCS** which then reduces at a relatively slower rate with the further increase in porosity. This is demarcated above 20% increase in porosity.

5. However the strength values are considerably good for filter applications with a maxima at 48.96 Mpa for a 15% porous body with zero surfactant. Porosity is exclusively on account of organic burnout.

4.6FLEXURAL STRENGTH:

TABLE 4.5

CODE	2.5 (7:3)	5 (7:3)	7.5 (7:3)	2.5-5 (7:3)	5-7.5 (7:3)	2.5 (1:1)	5 (1:1)	7.5 (1:1)	2.5-5 (1:1)	5-7.5 (1:1)
FS (Mpa)	**48.43**	**18.55**	**17.48**	**18.43**	**15.1**	**20.73**	**18.45**	**10.44**	**16.38**	**12.14**
+/-Err	**1.6**	**2.45**	**1.62**	**1.42**	**1.1**	**2**	**0.82**	**1.17**	**2.4**	**0.9**

FLEXURAL STRENGTH

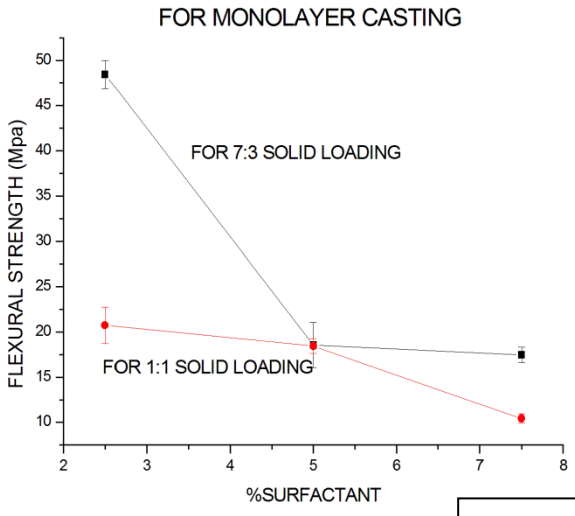


FIG 4.1.19

1. Flexural strength averaged, from (**table 4.5**) follows a similar analogy as that of CCS in Fig **4.1.19**the variation is rapid after an increase in porosity values beyond 20% Max value- **48.43**Mpa..
2. It has how ever been observed that in bilayer casted bodies fig **4.1.20** the variation in Flexural strength b/w two values is not much may be on account of a bilayer

behaviour when theindividual layers or layer with higher porosity restarins the other from expanding. Hence tensile yield is less. Max value-

3. Error margin more on account of varying porosity and its distribution, surface polishing also very vital, hence we need to have sensitive pretreatment of the surface to get better tensile properties

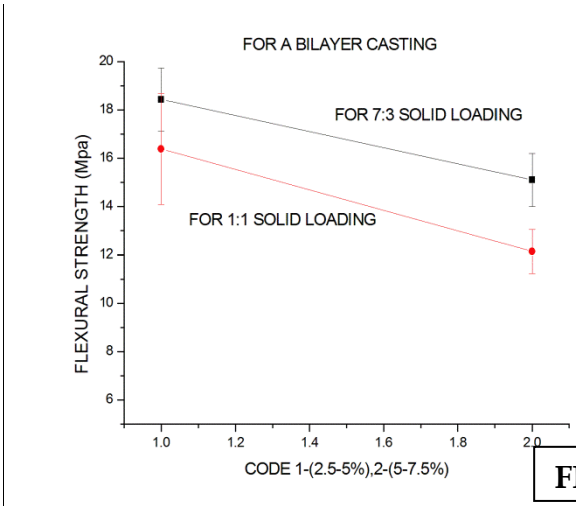
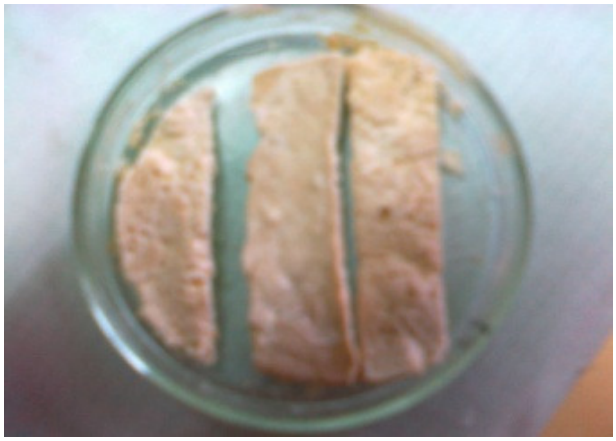


FIG 4.1.20

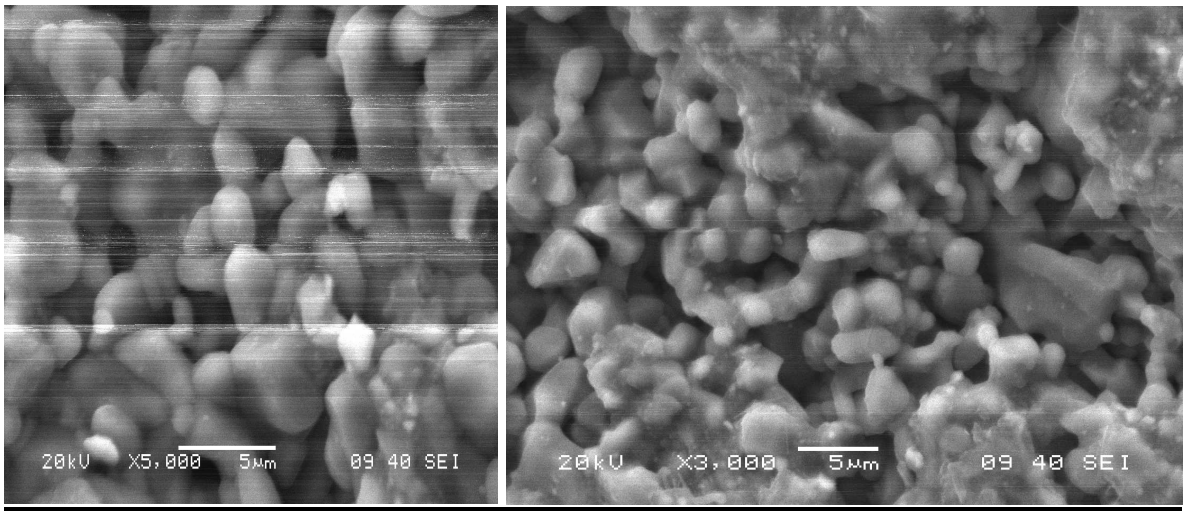
- A range of points couldn't be shown in the CCS plot on account of shaping complexity for testing as this has to be cut out from the casted material however the data points give us an tentative impression of the levels of variation encountered at different solid loading and surfactant concentration.



SCALY BODY THAT REGISTERED THE MAXIMUM POROSITY OF 60%.

4.7MICROSCOPY ANALYSIS:

SEM IMAGING OF A 24% POROUS GELCASTED ALUMINA



A **FIG 4.1.21** B

The above SEM images fig **4.1.21** show that the average size of pores lie around 5microns as shown by that extended tab line. This is the same sample that had been put to test in the Mercury porosimeter. The amount of porosity registered had a maximum pore size distribution of ***5 microns***.

Left portion or A subscripted image is representative of a relatively more magnified version of right subscripted image as B. From the above image we can infer that pore size distribution is still to be controlled this can however be achieved by a controlled addition of pore formers which is the easy way out else we can even control the stirring speed for surfactants but the method is critically difficult to monitor.

PICTURE TEMPLATES

GREEN GEL CASTED ALUMINA

A 2.5% SURFACTANT and 7:3 LOADED BODY

A 5% SURFACTANT and 7:3 LOADED BODY

SHOWS EXCELLENET GREEN MACHINABILITY.

EXPERIMENTAL SETUP TO SHOW FILTRATION

Fig A

Fig B

Fig C

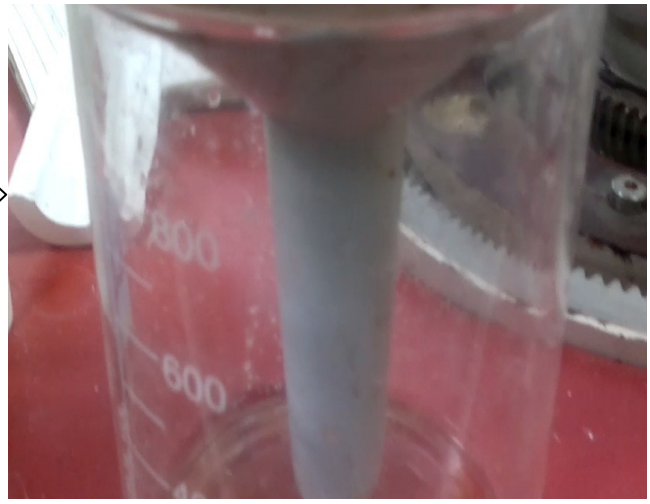


Fig D

Fig A and B are the practical demonstrations of the filtration process as evident in Fig A we can see the white filter candle made of gel casted multilayerd variably porous alumina body that has been manufactured and Fig B shows the collected filtered water quite contrary to the muddy water shown in Fig A, quite similarly we have a similar setup shown in Fig C and Fig D where the turbulence and mud content has been increased so it is but proved that filtration action is evidently clear and visible from the above figure.This is an evidence that the material has connected pores and can be used as a suitable filter material.

CONCLUSIONS

Finally Gel casting proved to be a successful endeavour with the set of reagents we have implemented into action. During the entire process framework a maximum of **60% porosity** was achieved at the laboratory scale, howsoever compressive strengths were a bit compromised with,the maximum compressive strength being **48.96 Mpa** for a 15% porous body which had only organic loading but for filter applications the compressive strength was impressive.There was a sudden reduction in CCS values when the porosity increased beyond 20%.Flexural Strength values owing to the poor tensile property exhibited by ceramics have been decent with the maximum valuing at **48.43 Mpa**. The minimum pore size achieved by surfactant entrapment lies around **5 microns** which deems it fit for microporous membrane functionality. Zeta potential values were the most stable at **0.5%** dispersant oncentration which itself took the pH value to 9.2 and gave a zeta potential of **-66.1 mV** which is strikingly impressive. Rheology values showed the best and most uniform thixotropic behaviour at **9:3** solid loading however on account of incorporating porosity in the material we reduced the solid loading to **7:3(alumina:water).** The average porosity for a **7:3 cast lied in between 26%-30% and the average porosity for a 1:1 cast lied in between 40-45%.**

This has a unique versatility over other filters as it can simultaneously behave as a water cum filter for corrosive liquids has other applications too. This process allows greater control over porosity distribution and pore size however the heating schedule including drying and firing needs to be uber critical as that holds the key to the perfectness of the material.

FUTURE WORK

We can still strive hard to gain control over the pore dimension and distribution as a continuation of this project, effect of pore formers in the entire scheme of things needs to be studied and analysed. Even more sensitive porous and multifunctional high end membranes can be designed by significantly controlling the porosity distribution and pore size. Different layers at different particle size of precursor material can be used, on grading size we can control their sintering and hence shrinkage leading to defect free gelcasted body which is pretty sensitive to heating schedules.

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